

August 1972

B72-10473

NASA TECH BRIEF

Langley Research Center



NASA Tech Briefs announce new technology derived from the U.S. space program. They are issued to encourage commercial application. Tech Briefs are available on a subscription basis from the National Technical Information Service, Springfield, Virginia 22151. Requests for individual copies or questions relating to the Tech Brief program may be directed to the Technology Utilization Office, NASA, Code KT, Washington, D.C. 20546.

Process for Synthesizing and Formulating Condensed Ring Polymers

A new method of synthesizing and formulating condensed ring polymers for use in moldings, laminates, and adhesives has been conceived with pyrrone polymers using a novel synthesis route.

Previous work with condensed ring polymers has involved the formation of long chain, high molecular weight, highly soluble precursor polymers having flexible bonds and highly polar functional groups. These polymers were subsequently converted to an insoluble, rigid, cyclized heteroaromatic structure by thermal treatment which eliminated one or more molecules of water from the chain per-monomer-unit. These volatiles have to be removed to obtain a cured resin with optimum properties. An example of this process is the preparation of pyrrone polymers which have been synthesized with polyamide acid amine precursors. These precursors have been made by the reaction of aromatic five-ring dianhydrides with bis(o-diamines), which are thermally or chemically cyclodehydrated to a heterocyclic ring system containing four or seven fused rings (depending on the dianhydride). The resulting polymer can have either a ladder or a semi-ladder structure, and an even more rigid backbone chain than the polyimides.

This new process forms low molecular weight, fully cyclized heteroaromatic prepolymers (or oligomers) under conditions which severely limit chain extension or branching. The oligomers are then more easily processed in conventional fashion with less volatile formation. The process is defined as follows:

- (a) A partially or fully cyclized prepolymer was prepared with controlled molecular weight by reacting a large excess of comonomer A with comonomer B. The average molecular weight and distribution were controlled by the ratio of the monomers, the higher the ratio, the lower the average weight and the narrower the distribution. To prevent branching and promote cyclodehydration, the reaction was conducted at an elevated temperature

and in dilute solution. It was also essential to maintain the desired ratio of monomers during the entire reaction in order to prevent chain extension and possible gelation. The prepolymer contained end groups associated with comonomer A.

- (b) The above procedure was repeated with an excess of B relative to A to produce a prepolymer with end groups for comonomer B.
- (c) Solutions of the two prepolymer oligomers (either with or without comonomers) were then mixed in as short a time as possible at a relatively low temperature and precipitated immediately. A powder was produced which was a molecular mixture of the two oligomers and which had a minimum of chain extension. Molecular mixing of the pyrrones at 450 K resulted in some chain extension although enough flow still remained to obtain good high pressure holdings.

The advantages of this process, as exemplified by pyrrone polymers, are several-fold. The volatiles generated during final cure are reduced (relative to the polyamide acid amine precursor) thus making the moldings substantially more void free. In addition the molded pyrrone polymer had a higher flexural strength and modulus (especially at 500 K and higher) and substantially improved oxidation resistance.

Note:

The following documentation may be obtained from:
National Technical Information Service
Springfield, Virginia 22151
Single document price \$3.00
(or microfiche \$0.95)

Reference:

NASA CR-1633 (N70-35716), Preparation and Characterization of Low DP End-Capped Pyrrone Moldings

(continued overleaf)

Patent status:

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457 (f)] , to the AVCO Corporation, Lowell, Massachusetts 01851

Source: C. T. Hughes and R. J. McHenry of
AVCO Corporation
under contract to
Langley Research Center
(LAR-10423)